Universitat Autònoma de Barcelona

2019/2020

Synthesis Laboratory

Code: 102530
ECTS Credits: 6

Degree	Type	Year	Semester
2502444 Chemistry	OB	3	2

Contact

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Use of Languages

Principal working language: catalan (cat)
Some groups entirely in English: No
Some groups entirely in Catalan: No
Some groups entirely in Spanish: No

Teachers

Marta Figueredo Galimany
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Jordi García-Antón Aviñó
Ona Illa Soler
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Prerequisites

As defined in the objectives, this subject is the experimental continuation of the theoretical subjects "Synthetic Methods" and "Chemistry of Coordination and Organometallic". The knowledge acquired on the subject "Structural Determination" will also be implemented. For this reason, it is highly recommended that students have passed or have completed these subjects during the first semester of the same academic year. On the other hand, as these theoretical subjects of the 3rd year are based on the knowledge acquired in the subjects of the 2nd course "Structure and Reactivity of Organic Compounds" and "Chemistry of the Elements", it is indispensable to have passed these last subjects to take the course "Synthesis Laboratory". It should be noted that the reactions that the student will perform and the compounds that he will synthesise and analyze form part of the contents of the aforementioned theoretical subjects and, consequently, in both the laboratory sessions and the evaluation of this course, these contents are given by well known.

Objectives and Contextualisation

The objective of this experimental subject is double. On one hand, to reinforce through a series of synthesis experiments and characterization of compounds, the understanding of the concepts developed in the subject "Synthetic Methods" and "Coordination and Organometallic Chemistry", which the student has completed in the first semester. On the other hand, being this the last experimental subject focused on chemical synthesis, it complements the laboratories that the student has study as part of the 2nd year subjects, "Structure and

Reactivity of Organic Compounds" and "Chemistry of Elements". In this way, the subject "Synthesis laboratory" provides the student with a solid formation in a good number of advanced synthetic techniques, of greater difficulty than those that are part of the laboratories linked to the 2nd year subjects.

The subject is constituted by a set of laboratory practices in which the student will synthesize and characterize a series of compounds. These have been selected with the aim of covering the largest possible number of synthetic experimental techniques and the widest variety of compounds, within the time available.

The synthetic training will be complemented by the use of some instrumental techniques (IR, UV and NMR spectroscopy) that students will use in the characterization of the compounds. In this sense, the knowledge acquired in the subject "Structural determination", taken during the first semester of the 3rd year, will be used. In addition, as in all laboratory courses, the learning of safety standards and the treatment of waste is also an essential part of the student's training. In particular, in this course, the student must become familiar with the handling of dangerous substances.

Competences

- "Interpret data obtained by means of experimental measures, including the use of IT tools; identify their meaning and relate the data with appropriate chemistry, physics or biology theories."
- Adapt to new situations.
- Apply knowledge of chemistry to problem solving of a quantitative or qualitative nature in familiar and professional fields.
- Be ethically committed.
- Communicate clearly in English.
- Communicate orally and in writing in ones own language.
- Develop synthesis and analyses studies in chemistry from previously established procedures.
- Evaluate the health risks and environmental and socioeconomic impact associated to chemical substances and the chemistry industry.
- Handle chemical products safely.
- Handle standard instruments and material in analytic and synthetic chemical laboratories.
- Have numerical calculation skills.
- Learn autonomously.
- Manage the organisation and planning of tasks.
- Manage, analyse and synthesise information.
- Operate with a certain degree of autonomy and integrate quickly in the work setting.
- Propose creative ideas and solutions.
- Reason in a critical manner
- Recognise and analyse chemical problems and propose suitable answers or studies to resolve them.
- Resolve problems and make decisions.
- Show an understanding of the basic concepts, principles, theories and facts of the different areas of chemistry.
- Show initiative and an enterprising spirit.
- Show motivation for quality.
- Show sensitivity for environmental issues.
- Use IT to treat and present information.
- Use the English language properly in the field of chemistry.
- Work in a team and show concern for interpersonal relations at work.

Learning Outcomes

1. Adapt to new situations.
2. Analyse situations and problems in the field of organic and inorganic chemistry, and propose answers or experimental studies using bibliographic sources.
3. Apply the acquired theoretical contents to the explanation of experimental phenomena.
4. Be ethically committed.
5. Characterise synthesised compounds using physical and spectroscopic methods.
6. Classify compounds by their most important reactivity characteristics: acid/base and oxidant/reductant.

7. Communicate clearly in English.
8. Communicate in English in the laboratory.
9. Communicate orally and in writing in ones own language.
10. Deduce the stable electronic configurations of an organometallic compound.
11. Describe basic safety regulations.
12. Determine the performance of a reaction.
13. Develop synthetic and analytic studies in the field of the organic chemistry from previously established procedures.
14. Develop the habits and skills of a laboratory.
15. Draft a laboratory logbook containing descriptions of the developed procedures, the observations made, the results obtained, the interpretation of the same and the conclusions.
16. Evaluate risks in the use of chemicals and laboratory procedures.
17. Follow safety procedures in the chemistry laboratory.
18. Follow standard laboratory procedures described in English.
19. Follow standard laboratory procedures.
20. Handle instruments to record different types of spectrums.
21. Have numerical calculation skills.
22. Identify the main functional groups in organic compounds and some of their reactions.
23. Identify the main reagents in a laboratory and their commercial presentation.
24. Identify the risks of synthetic reagents.
25. Innovate methods for adaptation to the interpretation of a specific molecular structure.
26. Interpret the safety notes on chemistry products.
27. Learn autonomously.
28. Manage the organisation and planning of tasks.
29. Manage, analyse and synthesise information.
30. Manipulate the main reagents and dissolvents in a chemistry laboratory.
31. Observe the physical and chemical properties of different substances.
32. Observe the reactivity and behaviour of representative compounds in the laboratory.
33. Operate with a certain degree of autonomy and integrate quickly in the work setting.
34. Order the most common ligands (or the ligands chosen as representative examples) by their properties (donor and/or accepter capacity, spectrochemical series).
35. Perform a synthetic and analytic study to determine chemical and physical properties using instructions supplied for a detailed procedure.
36. Perform correct evaluations of the health risks and environmental impact of magnetic fields.
37. Perform standard tests on which the results obtained are based.
38. Predict the reactivity of different organic functional groups under certain reaction conditions, as well as the structure of the products obtained.
39. Prepare an inorganic chemistry laboratory or experiment as described in English.
40. Prepare inorganic compounds with coordination bonds.
41. Prepare inorganic compounds with metal-carbon bonds.
42. Prepare solid state inorganic compounds.
43. Propose creative ideas and solutions.
44. Reason in a critical manner
45. Recognise potential risks in the laboratory before they are produced.
46. Recognise potentially dangerous reagents and dissolvents.
47. Recognise the electronic spectrums of coordination compounds.
48. Recognise the specific terminology of inorganic chemistry to communicate professionally.
49. Recognise the use of each reagent in the laboratory and take appropriate safety precautions in each case (special goggles and/or gloves, extractor hood, gas mask, etc.).
50. Relate bond theory and the models of inorganic chemistry with the reactivity of elements and their compounds.
51. Relate knowledge about the structure and reactivity of the elements and chemical compounds with their method or methods of obtainment and/or purification.
52. Resolve problems and make decisions.
53. Safely dispose of waste from chemical reactions.
54. Safely handle inflammable, toxic and/or corrosive reagents.
55. Safely handle the different radiations involved in each spectroscopic technique.
56. Safely handle the electrical circuits that form part of different spectrometers.

57. Selectively distinguish the rejection of reagents and chemical products.
58. Show initiative and an enterprising spirit.
59. Show motivation for quality.
60. Show sensitivity for environmental issues.
61. Summarise a report or article on inorganic chemistry in English.
62. Synthesise and purify a compound chemical.
63. Understand the labelling of chemical reagents in English.
64. Use IT to treat and present information.
65. Use data processors to produce reports.
66. Use graphic design programs to draw chemical formulas and their reactions.
67. Use safety equipment properly.
68. Use spectroscopy devices to confirm experimental results.
69. Use suitable strategies for the safe elimination of reagents.
70. Use the basic materials of a chemical laboratory.
71. Use the most common English chemistry terms.
72. Work in a team and show concern for interpersonal relations at work.
73. Work safely in the laboratory while following the adequate procedure.
74. Write simple laboratory reports in English

Content

Block I
P1. Synthesis of 2-acetylcyclohexanone from cyclohexanol
P2. Wittig Reaction: Synthesis of (Z)- cinnamic acid
P3. Hydroboration reaction of an alkene (anti-Markovnikov hydration)
P4. Synthesis of 2-oxyranyl-1-phenyletanol from Benzaldehyde
P5. Synthesis Project

Block II
P1. Co (III) complexes. UV spectroscopy. Isomerization of coordination. IR spectra.
P2. Co(salen) preparation. Reaction with O2
P3. Preparation of phenylmagnese bromide and triphenylphosphine
P4. Preparation of ferrocene
P5. Preparation of metal-carbonyl complexes. IR and NMR spectroscopy

Methodology

This course is structured in 24 laboratory sessions of 4 hours each. These sessions are divided into two blocks of 12 sessions each. Block I corresponds to the synthesis of organic products, while block II focuses on the preparation of inorganic products.

Prior to the beginning of the laboratory sessions, there will be an informative session (compulsory attendance) which will explain to the students the operating rules of these laboratories, the security measures that they will have to follow and some fundamental aspects of the practices that they have to perform

There will be written tests to evaluate:

The degree of understanding and knowledge that the student has achieved by reading the practice script, and/or the related theoretical aspects that accompany the practice (all available as material in the Moodle virtual platform), and/or the practical performing of the experiments.

These tests will be the most important element in the student's qualification.

Activities

Title	Hours	ECTS	Learning Outcomes

Type: Directed			
Laboratory Presentation	4	0.16	9, 58, 59, 11, 12, 53, 57, 63, 28, 29, 24, 26, 71, 54, 4, 60, 33, 43, 44, 46, 49, 48, 45, 51, 17, 18, 72, 67, 16
Laboratory Sessions	96	3.84	1, 2, 3, 27, 5, 6, 8, 7, 9, 10, 58, 59, 11, 14, 13, 12, 53, 57, 63, 36, 74, 28, 29, 22, 23, 24, 25, 26, 71, 20, 56, 55, 54, 30, 4, 60, 32, 31, 33, 34, 38, 41, 40, 42, 43, 44, 37, 35, 39, 47, 46, 49, 48, 45, 15, 51, 50, 52, 61, 17, 19, 18, 62, 21, 73, 68, 70, 64, 69, 67, 66, 65, 16
Type: Autonomous			
Preparation of the experiments	45	1.8	2, 27, 6, 7, 9, 10, 58, 59, 11, 28, 29, 22, 24, 25, 26, 71, 4, 33, 34, 38, 43, 44, 37, 46, 49, 48, 45, 51, 50, 61, 21, 72, 64, 66, 65, 16

Assessment

The student will obtain a mark from each of the two blocks: Inorganic and Organic Chemistry.

The mark of each block will consist of two parts:

Part 1: Final Exam (Contribution to the final mark in the subject = 40%)

It consists of a written test of evaluation that will consist of 2 parts (Inorganic and Organic Chemistry). A second test will be scheduled for the sole purpose of retaking this part or grading the mark up.

Part 2: Others (Contribution to the final mark in the subject = 60%)

In this part, different aspects will be valued: brief written tests of evaluation (prelabs), results of the experiments, the book, realization of the project (part Q. Organic) and the attitude in the

laboratory). No retakes are programmed for this part.

Qualifications of the Subject:

Student who PASSES the subject:

Student who meets all of the following requirements:

a) The final exam has obtained a score equal to or greater than 5 to each of the parts that comprise it (Q. Inorganic and Q. Orgànica).

b) Student with a global grade of each block equal to or greater than 5.

Student who DOES NOT PASS THE SUBJECT:

Student who does not meet any of the requirements indicated in the "Students that passes the subject".

OUTSTANDING MENTION:

The Outstanding Mention may be awarded to students who have obtained a grade equal to or greater than 9.00. Their number may not exceed 5% of the students enrolled in a subject (groups of morning + afternoon) in the same corresponding academic course.

NOT ASSESSABLE Student:

Any student who has one or more lack of assistance without justification. In no case, the lack of assistance may exceed more than one session (4h) per Bloc. Any lack of assistance must always be duly justified immediately to the responsible Professor.

RETAKE:

To retake this subject, the students must have been previously evaluated in a set of activities whose weight equals to a minimum of two thirds of the total grade of the subject.

The retake of all assessment activities carried out in the Laboratory sessions is not contemplated.

The retake exam will be retrieved also through a written test.

IRREGULARITIES IN EVALUATION TESTS

Without prejudice to other disciplinary measures deemed appropriate, the irregularities committed by the student that can lead to a variation in the qualification of any assessment activity will be qualified with a zero. Therefore, copying, plagiarizing, cheating, etc. In any of the assessment activities it will imply failing it with a mark equal to zero.

SECURITY WARNING IN THE LABORATORY

Any student involved in an incident that could have serious consequences of security may be expelled from the laboratory and fail the subject.

Assessment Activities

Title	Weighting	Hours	ECTS	Learning Outcomes
Assessment of the student's skills and attitude in the laboratory. Correction of the laboratory book. Design of a steps synthesis.	20	0	0	1, 2, 27, 58, 59, 14, 53, 74, 28, 23, 20, 56, 55, 54, 30, 60, 32, 33, 35, 39, 46, 49, 15, 52, 17, 18, 73, 72, 68, 70, 64, 69, 67, 66, 65
Written tests	80	5	0.2	3, 5, 6, 8, 7, 9, 10, 11, 13, 12, 57, 63, 36, 29, 22, 24, 25, 26, 71, 4, 31, 34, 38, 41, 40, 42, 43, 44, 37, 47, 48, 45, 51, 50, 61, 19, 62, 21, 16

Bibliography

BLOCK I

Theoretical Concepts:
Organic Chemistry
Jonathan Clayden, Nick Geeves, Stuart Warren 2nd Edition, 2012
ISBN: 978-0199270293 Oxford University Press

Experimental Part:
Experimental Organic Chemistry
Laurence M. Harwood, Christopher J. Moody, Jonathan M. Percy 2nd Edition, 1999
ISBN: 0-632-04819-0 Blackwell Publishing

Vogel's Textbook of Practical Organic Chemistry
A.I. Vogel, A.R. Tatchell, B.S. Furnis, A.J. Hannaford, P.W.G. Smith 5th Edition. Prentice Hall, 1996. ISBN: 0582462363

BLOCK II

Theoretical Concepts:
Shriver-Atkins, Química Inorgànica, 4ª edició, Ed. McGraw-Hill, 2008
C. E. Housecroft, A. G. Sharpe, Química Inorgánica, 2ª edició, Ed. Pearson, 2006 (capítols 19, 20, 23, 25 i 26)
D. Astruc, Química Organometàl·lica, Ed. Reverté, 2003

Experimental Part:
G.M. Williams, J. Olmsted III, A. Breksa III, J. Chem. Educ. 1989, 66, 1043.
R.B. Penland, T.J. Lane, J.V. Quagliano, J.Am. Chem. Soc. 1956, 78, 88.
T.G. Appleton, J. Chem. Educ.1997, 54, 443.
P. Simpson, Compuestos organometálicos de elementos de grupos principales, Ed. Alhambra, 1973, 120-123.
F.R. Hartley, G. Temple-Nidd, Educ. Chem. 1975, 12, 6.
P.W. Wiggins, Educ. Chem. 1973, 10, 52.
R.J. Angelici, J. Chem. Edu. 1968, 45, 119.